Analysis of Vermiculite & Vermiculite Containing Products

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There is currently a lot of attention being paid to analysis of Vermiculite and products containing Vermiculite. Vermiculite is a naturally occurring mineral composed of shiny flakes that resemble mica. Vermiculite when heated expands into wormlike accordion shaped pieces up to 30 times their original size. Because of all the air pockets created when this expansion occurs the material is a great thermal insulator.

One of the most productive Vermiculite mines was located in Libby Montana. Gold miners discovered vermiculite at the mine in 1881. In the 1920s the Zonolite Company formed and began mining it. The vermiculite at the mine was ground down to a specific size (beneficiation) depending on its final intended use, and then puffed up or expanded in a furnace (exfoliation). The final product was packaged and sold as Zonolite insulation. This product was distributed mostly by rail, all across North America and ended up in the attics of millions of homes as Vermiculite Attic Insulation (VAI). No one knows exactly, but it is estimated that up to 35 million homes in the U.S. alone contain VAI. W.R. Grace purchased the Libby mine in 1963 and greatly increased its production until 1990, when it was closed. Prior to its closing much of the world’s supply of vermiculite came from this mine. It was the source of over 70% of all vermiculite sold in the U.S. from 1919 to 1990.

Because of its properties as an insulator and because it is extremely lightweight, chemically resistant and also fireproof, Vermiculite ended up as a constituent in many different building materials. It also worked very well as a carrier for chemicals in fertilizers. Vermiculite was used in its pure form as packing material, attic insulation, block fill insulation in foundations. It was used as a constituent in insulating and fireproof boards and molded shapes for furnaces and fireplaces, sprayed on fireproofing, acoustic panels, brake linings, various cement and concrete mixtures, plasters, soil conditioners, seed germination, chemical spill kits, etc.

Unfortunately the Libby mine had a natural deposit of asbestos running through it which contaminated the vermiculite product. The current concern over vermiculite and vermiculite containing materials (VMCs) is not the vermiculite itself but rather the asbestos that may be present as an accessory mineral or contaminant. The EPA along with the Agency for Toxic Substances and Disease Registry (ATSDR) initiated a national consumer awareness program in 2003 to educate the public on the topic of asbestos in Vermiculite Attic Insulation (VAI). Asbestos contamination can be found in vermiculite from other mines however Libby was unique in the amount of asbestos present. Vermiculite is currently mined at three other US locations and abroad and has relatively low levels of asbestos contamination.

The primary concern with asbestos is inhalation which can lead to lung diseases such as asbestosis, lung cancer and mesothelioma. The asbestos found in loose vermiculite arguably poses a greater risk since it is not bound in the matrix as with other building materials.

ANALYTICAL APPROACHES

As stated earlier, the vermiculite itself is typically not the target analyte but rather the asbestos that may be present as a contaminant. The vermiculite actually obstructs that analysis. For various reasons Vermiculite represents a problem matrix for asbestos analysis by microscopy. One reason is that since the size of the vermiculite plates can be orders of magnitude larger...
Analysis of Vermiculite—continued

than the asbestos we are looking for, it gives smaller asbestos fibers and bundles plenty of places to hide. In some samples, larger rice grain sized bundles of asbestos are present and can even be seen to the naked (trained) eye upon careful examination.

Another issue with asbestos analysis of loose fill vermiculite is that the asbestos is generally not distributed uniformly throughout which makes the task of obtaining an accurate percentage difficult, if not impossible.

**Cincinnati Method (EPA/600/R-04/004)**

Early on the EPA suggested this method for determining the asbestos content of Vermiculite. It took advantage of the fact that the larger bundles of asbestos would rapidly settle out in water (aka “sinks”) leaving the Vermiculite floating on the surface. In a sense, this approach was an elegant way to separate the wheat from the chaff. After settling the vermiculite is skimmed off the top and discarded. The suspension is aspirated off and saved for optional subsequent TEM analysis leaving the “sinks” to be dried and analyzed by Polarized Light Microscopy (PLM). It starts with a rather large sample (a kilogram is collected and sent to the lab) and if the large bundles were present, the technique provided a good means of concentrating any asbestos present for detection by light microscopy. If only fine fibers are present they may go undetected with this method unless the optional TEM analysis is performed on the water wash. The TEM analysis provides results expressed as fibers per gram and this can be of limited value to the end user trying to apply an action level expressed as a percentage. This method is rather time consuming and laborious (and therefore relatively expensive) for routine analysis and so was under-utilized.

**EPA 600/R-93/116**

As an alternative to the Cincinnati method many are simply leaning back on the standard EPA 600 PLM analysis. However the presence of large vermiculite books can hinder a proper monolayer slide prep for analysis (necessary for proper quantitation) as well as obscure asbestos fibers.

**EPA 600/R-93/116 with milling (aka CARB 435)**

As a means to address the large particle size and inherent heterogeneity of the vermiculite, the sample can be lightly milled to reduce the particle size prior to prep and analysis. This type of matrix modification is described in the EPA 600/R-93/116 PLM method. Though many think of the California Air Resource Board (CARB) Method 435 when milling is mentioned, there is nothing truly unique about this method. It is a PLM 400 point count with milling prior to further prep and analysis. The EPA PLM analysis method is much more comprehensive than that of CARB 435 and outside of California the end user may want to stand behind results derived from an EPA method. The downside of the milling approach is that all of the non-asbestos material is now present in the slide prep along with any asbestos that may be there as well. The upside is that this can actually aid the analyst in the determination of the true percentage of asbestos in the sample.

**New York State Method 198.8**

New York State has made Vermiculite and Vermiculite Containing Materials (VCMs) a main issue since 2012. New York State currently mandates that any and all loose fill vermiculite be considered Asbestos Containing Material (ACM). No analysis necessary, no analysis allowed, and no analysis results will be entertained, to consider this material anything other than ACM. A few other states and Canadian provinces presume loose fill vermiculite to be ACM but do not disallow analysis to prove otherwise. Beyond loose fill vermiculite, NY also now has special analysis rules for building materials (other than sprayed-on fireproofing) containing greater than 10% vermiculite (think textures and popcorn ceiling). These materials must undergo gravimetric reduction prior to PLM analysis via the NYS ELAP 198.6 Method. As per the latest NYS ELAP guidance, the following disclaimer must accompany these results: “This method does not remove vermiculite and may underestimate the level of asbestos present in a sample containing greater than 10% vermiculite.”

And finally, for spray on fireproofing containing any amount of vermiculite (SOF-V), New York has created a new method designed specifically for this material type, New York ELAP method 198.8. This method, even more labor intensive than the Cincinnati method, is a two stage analysis. The first stage involves gravimetric reduction by ashing in a muffle furnace, acid treatment and then painstaking removal of any floating vermiculite prior to a PLM analysis for Chrysotile Asbestos. If >1% asbestos is detected analysis is terminated, otherwise a second phase of prep and analysis is initiated. This second phase involves gravimetric reduction again but this time by heavy liquid separation. The ashed and acid treated residue is dispersed in Lithium Meta-Tungstate, a liquid that is mixed with fiber free water to obtain a liquid with a density between vermiculite and amphibole asbestos. When the sample is centrifuged at
Analysis of Vermiculite—continued

approximately 3600 rpm, the vermiculite rises to the top and the amphibole settles to the bottom of the centrifuge tube. This is a labor intensive process involving multiple washes and re-centrifugations. After recovering the final resulting pellet of material (density greater than 2.75 g/cc), it is re-suspended and then filtered prior to analysis by PLM. The percentage asbestos (if any) by PLM analysis is combined with the gravimetric results to calculate the asbestos percentage in the original sample. In our lab’s experience, after hundreds of analyses, the only samples determined to be ACM (>1%) were heavily loaded with Chrysotile which arguably would have been detected with “regular” PLM.

A Common Sense Approach

The ASTM D22.07 asbestos committee has a Draft TEM Qualitative Method for Loose Fill Vermiculite currently in the balloting stage. This method incorporates a simplistic water wash like the Cincinnati method but rather than focus on the sinks this method filters a portion of the suspension and looks for asbestos fibers by TEM. The logic here is that if asbestos is present in either the sinks or the floats after aggressive agitation in water asbestos should be detected in the water. Two rounds of round robin analyses among multiple labs have produced very encouraging results. Though this method is still in Draft form, many labs are already offering TEM qualitative as an analytical option. As the name implies this method is a presence or absence test. Once asbestos is identified analysis is terminated and no attempt is made to quantify the asbestos that is there. This makes sense on several different levels. Firstly, it is well documented that asbestos, if present, is generally not homogeneously distributed throughout loose fill vermiculite. Why extend analytical effort trying to nail down an accurate percentage when samples collected adjacent to one another may have different asbestos percentages? And if the asbestos that is present is identified as one of the types present in Libby vermiculite (the Libby Amphiboles) we have a pretty good idea as to the range of asbestos that can be present as well as the potential danger. The other point to make is that we know that 1% is not a health based limit even for regular building materials. The asbestos present in vermiculite is not bound up like it typically is in a building material and therefore the risk is potentially greater, even at percentages well below 1%. Unfortunately the 1% rule still predominates and often times a Qualitative result will not suffice no matter how much sense it makes.

When you must have an asbestos percentage associated with a set of Vermiculite or VCM samples then it is important to note that in a study conducted by EMSL and involving multiple independent labs across the country, the method that performed best for loose fill vermiculite was EPA 600/R-93/116 PLM method with milling. For Vermiculite Containing Materials (VCMs) represented by spray on fireproofing, the method that performed best was the EPA 600/R-93/116 method with gravimetric reduction.

Financial Relief

If Libby vermiculite is installed in a building you own or rent you can possibly get financial relief from removal costs through the Zonolite Attic Insulation Trust funded by W.R. Grace. The webpage http://www.zonoliteatticinsulation.com/ contains all the information you need to submit a claim for reimbursement. The Trust may provide a reimbursement contribution of 55% of the abatement cost for eligible Claimants up to a ceiling of 55% of a $7,500 removal bill (or $4,125). For example, if you spent $7,500 removing and replacing the insulation, you are potentially eligible for the maximum reimbursement of $4,125. If you spent $3,000 for abatement and re-insulation, you are potentially eligible for reimbursement of $1,650 (55% of $3,000). Ironically the determination of whether or not your vermiculite is from Libby is made by its Barium content not its asbestos content! This approach stems from a published study by Gunter, et al. If the Barium concentration is determined to be >1500 ppm by ICP (Inductively Coupled Plasma) analysis then the trust assumes the vermiculite is Libby sourced and funds are released. To date approximately 90% of samples tested by EMSL have exceeded the action level. This statistic is not surprising knowing that Libby produced so much of the vermiculite used in this country for close to 70 years.

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EPA published the “Purple Book” (Guidance for Controlling Asbestos-Containing Materials in Buildings) in 1985, and it has not been updated since. In 2010, the EPA asked the Environmental Information Association to create an update for the guidance manual, and EIA is proud to announce that it will be available at the EIA 2015 Conference and Exhibition in Atlanta.

Led by Tom Laubenthal, the distinguished experts on the Purple Book Committee have produced an outstanding document to replace EPA’s outdated Purple Book. This tool is essential to the asbestos industry, especially to building owners and managers, and anyone who works in our field should have a copy. You will be able to preview and order our own copy of the EIA Purple Book at the EIA Conference and Exhibition, at the Grand Hyatt Atlanta in Buckhead.

We look forward to seeing you at EIA 2015 in Atlanta!